

**GENERAL
ENGINEERING
LABORATORY**

N68 18033

Code-1

**PROGRESS
REPORT**

**DEVELOPMENT OF LOW TEMPERATURE
DIELECTRIC COATINGS
FOR ELECTRICAL CONDUCTORS**

BY

K.N. MATHES

OTS PRICE

XEROX \$ *1.60 ph*
MICROFILM \$ *0.80 mf*

OCTOBER 15, 1962

RKT 611

GENERAL  ELECTRIC

Fifth Quarterly Report
October 15, 1962

Development of Low Temperature Dielectric Coatings
for Electrical Conductors

Contract No. NAS8-2442
Control Number: TP85-498(CPB-02-1096-61)
(W/O #8500-0-0224-000-8500-11-431)

General Electric Company
Missile and Space Vehicle Department
Philadelphia, Pa.

Report Prepared by:

K. N. Mathes
General Engineering Laboratory
General Electric Company
Schenectady 5, N.Y.

Report Prepared for:

George C. Marshall Space Flight Center
Huntsville, Alabama

Fifth Quarterly Report
October 15, 1962

DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS
FOR
ELECTRICAL CONDUCTORS

INTRODUCTION

The development and evaluation of magnet and hook-up wire with superior properties at cryogenic temperatures have been described in the fourth quarterly and final report dated July 16, 1962.

The contract extension includes evaluation of the newly developed wires (with heavy polyvinyl formal enamel, extruded polyvinylchloride, and extruded polytetrafluorethylene as controls) at cryogenic temperatures after thermal aging at 250°C, at 120°C and at 120°C in vacuum. Exposure at high humidity is also to be included. This report describes progress in setting up these aging programs.

Contract work includes also the development of flat ribbon cable for application at cryogenic temperatures and the electrical evaluation of the liquid cryogenic gases as well.

SUMMARY AND CONCLUSIONS

Wire Evaluation

It is possible to obtain ML coated wire from at least two sources (General Electric and Phelps Dodge) with excellent flexibility at liquid helium temperature. Several satisfactory samples have been obtained from General Electric--the last with only a single factory operation involving multiple passes through the baking oven. The General Electric wire will be used in the aging program. All of the other wires are in hand and most of the samples for the aging programs have been prepared.

Electric breakdown in liquid hydrogen has been measured for a variety of wire insulators with results summarized in Table I. The breakdown in liquid hydrogen is comparable with that in liquid nitrogen and generally higher than in liquid helium.

Thermal Aging

Wire samples are being aged at 250°C in air and will be removed after 60 and 120 days for evaluation.

Facilities for aging at 120°C in vacuum have been completed and a short trial run is underway. A vacuum of 10^{-4} torr or somewhat better can be held. As soon as reliable vacuum performance is assured both air and vacuum aging of wire samples at 120°C will be started.

New Wire and Cable Constructions

A parallel conductor, flat ribbon cable (obtained from NASA-Huntsville) both cracks and delaminates when bent repeatedly about a 1/2 inch mandrel in liquid helium but does not fail over one-inch mandrel if stress relieved. Since the Mylar (Dupont's polyester film) was expected to have limited cold flexibility, attempts are being made to obtain a similar ribbon cable made with either polytetrafluorethylene or more likely with polyfluorethylene propylene (FEP) film.

Arrangements are under way also with the Film Department of Dupont to obtain polyimide (H) film with a coating of FEP resin from which paralleled conductor ribbon cable can be made. Such a construction is expected to have superior flexibility at cryogenic temperatures and stability in liquid oxygen.

For comparison Dupont will supply samples of wire insulated with taped coatings of polyimide (H) film bonded with FEP resins.

Evaluation of Cryogenic Liquids

Completely redesigned equipment for sphere gap breakdown in cryogenic liquids has been constructed (See Figs. 2 and 3). The new equipment has a number of advantages but most important will permit evaluation of very pure materials.

The dissipation factor and capacitance of the liquid nitrogen and helium have been measured at several frequencies. It is difficult to account for dimensional changes. Consequently the accuracy of the dielectric constant measurement is in question but compares quite well with the values in the literature. Absence of appreciable polarization for both nitrogen and helium is indicated by the constancy of the dielectric constant and the very low values of dissipation factor.

It appears possible to design suitable liquid cells for the accurate measurement of dielectric constant at cryogenic temperatures. The considerable expense involved does not seem to be justified in this program.

OBSERVATIONS AND SUMMARY OF TEST RESULTS

Wire Evaluation

Flexibility

Variability discovered in the cold temperature flexibility of polyimide (Dupont ML) insulated wire resulted in several attempts to improve wire quality and uniformity. By using slower speeds and higher oven temperatures the GE wire section in Schenectady, after several trials, has been able to make satisfactory ML coated wire with a single operation, using the usual number of multiple passes through the wire dies and baking oven. Previous samples had been given extra passes through the baking oven in order to obtain an adequate bake. The latest sample does not fail under repeated, reverse bends over a 1/8 inch mandrel in liquid helium (see below). It is also more uniform in color.

Since a sample of larger wire with Triple ML insulation from Phelps Dodge (Fort Wayne) became available it was evaluated also. As a result heavy ML insulated wire was obtained also from Phelps Dodge which passed the 1/8" mandrel test. It was light and very uniform in color. A triple ML coating is also on order from Phelps Dodge but no shipping promise can be obtained since it is a non-standard product.

Since ML coating on copper performs so well at cryogenic temperature, samples of felted asbestos (without inorganic binder) coated with ML polymer were obtained from the GE Wire and Cable Department at Lowell, Mass. The ML polymer did not appreciably penetrate the asbestos and instead tended to film on top. It tended to crack more readily than the film on copper but may not have been baked to an optimum degree. Although interesting, this wire insulation will not be included in the aging program.

Sufficient samples of the three cryogenic insulations - heavy ML, heavy ML plus aluminum phosphate - felted asbestos and aluminum phosphate - felted asbestos are available for the thermal aging programs and have been evaluated as reported previously. New samples of heavy polyvinylformal (GE Formex), extruded polytetrafluorethylene (Surprenant) and thin wall extruded polyvinylchloride (Surprenant) have been obtained to serve as control wires. The recent flexibility results on these as well as the new developmental wires are included in the table below.

<u>Wire (#22) Insulation</u>	<u>Wall in.</u>	<u>Performance - 10 reversebends in Liquid Helium</u>
Heavy Formex	.0012	Failed 1/2", OK 3/4" mandrel
Extruded Teflon	.012	Failed 3/4" mandrel
Heavy ML (GE-single operation)	.0014	OK - 1/8" mandrel
Triple ML (#16 wire-Phelps Dodge)	.0010	OK 1/4" mandrel - too stiff to be bent about a 1/8" mandrel
Heavy ML (Phelps Dodge)	.00185	OK - 1/8" mandrel
Extruded PVC (thin wall)	.0045	Failed 1-3/4" mandrel(single bond)
Felted asbestos-ML overcoat (without inorganic binder)	.0050	Asbestos, itself, did not separate even on 1/8" mandrel. ML overcoat spalled off on 1/8" mandrel and exhibited radial cracks on 1/4" mandrel but was OK on 1/2" mandrel.

Electric Breakdown

In the fourth and final report dated July 15, 1962, breakdown voltage is compared for samples in liquid nitrogen, in liquid helium, and in vacuum at liquid helium temperature (Fig. VI). The low values in liquid helium particularly for spacer type insulations like asbestos are attributed to the poor electrical breakdown of the liquid helium itself. It is, of course, possible that the very low temperature might be involved. Because of this possibility and the interest in liquid hydrogen itself, breakdown measurements of NEMA twisted pair samples immersed in liquid hydrogen have been made. These results are compared with the previous results in Table I. Additional results for some newer wire samples are included also.

TABLE I.

AVG. BREAKDOWN IN VOLTS AT 60 CYCLES

<u>WIRE INSULATION</u>	<u>GAS</u>	<u>LIQUID</u>			<u>VAC</u>
	<u>AIR</u>	<u>NITROGEN</u>	<u>HYDROGEN</u>	<u>HELIUM</u>	<u>VACUUM</u>
(Temperature)	(296°)	(77°K)	(20°K)	(4°K)	(4°K)
Heavy Formex	5500	8900	8200	6970	7830
" " (new spool)	-	-	-	8000	-
Heavy Alkanex	9230	9770	9430	8230	8270
Heavy ML (Exp.#568)	10250	9070	8970	7830	7530
" " (Com'l plus 2 rebakes)	6330	6170	6800	6130	5780
" " (single operation)	-	-	-	9030	-
Triple ML (on #16 gauge wire)	-	-	-	11170	-
Teflon	6070	7700	8230	6300	7400
Nylon (Fused Fiber)	3400	4100	4700	3400	4300
Asbestos (plus phos- phate binder)	1000	3770	3270	2000	3470
" (over HML)	5000	7870	7330	5970	5570
" (" " plus HML overcoat)	5500	8800	9770	3120	7670

It is apparent that the breakdown values in liquid hydrogen are comparable to those in liquid nitrogen within the variation that is expected for such breakdown tests. It is interesting that higher values apparently were not obtained which might have been expected since the breakdown voltage of liquid hydrogen was shown in the earlier work to be appreciably greater than that for liquid nitrogen.

Aging

Wire insulations for the thermal aging program include the following:

Heavy ML (new G.E.)
Phosphoasbestos impregnated, felted asbestos
Above over heavy ML

Aging (Continued)

For control - Heavy Formex
 Extruded PTFE (Surprenant Teflon)
 Extruded PVC (Surprenant - Thin Wall)

Samples for electric breakdown, flexibility, dissipation factor, capacitance and insulation resistance have been prepared and are being aged at 250 C. One group of samples will be removed after 60 days and a second group after 120 days. The position of the samples in the oven will be rotated periodically even though the oven temperature is quite uniform (controlled within $\pm 2^{\circ}\text{C}$) and adequate ventilation with fresh air bleed is assured.

Vacuum facilities for aging at 120°C have been constructed (see Photographs 4 and 5). The samples are contained in 6" diameter pyrex tubing with a 3/4" port connected to the vacuum system with an "O" ring compression coupling. A mechanical forepump is followed with an air-cooled diffusion pump to obtain vacuum. A thin wall, stainless steel "U" tube with a copper tube at the bottom is used as a trap between the samples and the pump. The trap is immersed in 5 foot dewar containing sufficient liquid nitrogen to last over weekends and short holidays. A continuous vacuum of better than 10^{-4} torr exceeds the contract requirement of 10^{-3} torr but does not reach the desired value of 10^{-6} torr. Considerable effort has been expended in efforts to reach the better vacuum. All parts of the system have been checked satisfactorily with helium leak detection equipment. The system has been carefully washed and baked to overcome outgassing to some extent. Vacuum consultants are of the opinion that a larger diffusion pump would not help unless the vacuum lines and ports were increased in size. Such expense does not seem justified.

A trial run of some but not a complete sample array is in progress to prove out the vacuum system. The test will be concluded after about 2 weeks if satisfactory performance continues. The final vacuum run will then be started along with samples which will be aged in air at the same time. Arrangements are being made to shut down the ovens automatically in case of vacuum failure so that the samples in vacuum will not be damaged in the case of such failure. Such care is important since considerable time and expense is involved.

New Wire and Cable Constructions

The contract extension includes a phase devoted to the development of parallel conductor, flat ribbon cable, to the extent that time and funds are available. While the main effort is now being expended in the aging studies, preparation for development of cryogenic flat ribbon cable is being started. As a first step, a small sample of polyester film (Dupont's Mylar) insulated cable (Methode Plyoduct PD812 P4 - 1 inch wide; .012 in. thick - contains 12 copper strips which are 1 mm wide, .0055 in. thick and spaced 1 mm apart), now under investigation at the George C. Marshall Space Flight Center, Huntsville, has been supplied by the contracting officer. Mandrels like those used for the repeated flexibility wire test have been adapted to clamp the ribbon sample so that it can be wound back and forth between them. In so doing it is necessary that after the first turn the ribbon conductor must wind over itself. It was found that failure will occur even on large mandrels at the split clamp at cold temperatures unless the samples are first wound back and forth several times at room temperature. Initial strains appear to be removed in this way but the mechanism is not clear.

Tests have been made with 1 inch, $\frac{1}{2}$ inch and $\frac{1}{4}$ inch mandrels. If stress relieved as described above no failure is obtained in liquid helium with the 1 inch mandrel. However, both delamination and cracking occur with the $\frac{1}{2}$ and $\frac{1}{4}$ inch mandrels (see Photo #1) - the delamination occurs first after several bends.

It was recognized at the start that Mylar has limited flexibility at cryogenic temperatures. Attempts are being made to obtain from several sources ribbon cable made with polyfluorethylene propylene (FEP Teflon) film. These manufacturers are also being asked to supply samples of other films which they may suggest. These will be screened for flexibility in liquid helium.

Because the new polyimide film (Dupont H film) is known to have excellent flexibility at cryogenic temperatures, excellent stability in liquid oxygen (NASA tests) and superior resistance to atomic radiation, it would appear to be an excellent candidate. H film is not yet available commercially (expected late 1963) so that arrangements are being made to supply film to one ribbon cable manufacturer (not yet selected) for trial. These samples will include some coated on one side with a thin FEP film to provide bonding between films and the encased parallel conductors. However, other bonding techniques for the H film will be tried also.

Samples of round hook-up wire have already been made using H film taped over the conductor in several ways and bonded with FEP film by heat-sealing techniques. Samples of such wires are being supplied by the Dupont Co. In this way it will be possible to compare ribbon with wrapped, round wire construction. It should be noted that polyester (Mylar) wrapped wire was evaluated early in the program and found to have limited flexibility. The results with Mylar ribbon cable indicate that it is superior to round wire and that the ribbon conductors may be particularly suitable for cryogenic use.

Evaluation of Cryogenic Liquids

Eccentricity of the spheres in the device used in earlier work to obtain breakdown in cryogenic liquid is believed to have caused errors in some cases, particularly with small springs between the spheres. T.J. Lewis at Queen Mary College, London, England, has found that impurities affect the impulse breakdown voltage of liquid argon and nitrogen. It is probable that solid particles of oxygen and nitrogen (from air) dispersed in liquid helium (and hydrogen) may adversely affect the breakdown values. The relatively high values of the dielectric constants for solid air particles as compared to liquid helium may be important in this respect. For practical as well as theoretical reasons it seems wise to investigate the effect of impurities in electric breakdown.

In consequence, new sphere-gap breakdown equipment shown in Photos 2 and 3 has been constructed. The new device has several advantageous features.

The inner chamber can be evacuated and

- (a) a pure gas can be admitted under the vacuum so that impurities can be avoided. The pure gas can then be liquified in place.
- (b) Temperature control can be attained and relatively low temperatures produced by using a vacuum in the outer chamber.
- (c) The device has been designed to accommodate standard cryostat headers.

- (d) The advantages of the previous device have been retained - steel ball bearings held by magnets, etc.
- (e) The mechanical support has been designed to decrease eccentricity as micrometer adjustments are made from the top (outside).
- (f) Breakdowns can be observed visually from the outside through the glass envelopes.

As soon as possible new measurements will be made particularly on super-cooled liquid helium in an attempt to determine if the low breakdown voltage measured earlier is intrinsic or a result of impurities, boiling, etc.

In the last quarter, capacitance and dissipation factor measurements have been made on liquid helium and nitrogen. In the first measurements a simple unguarded, concentric stainless steel cylindrical cell was used. Measurements were made just above and in the liquid. Unfortunately the losses and the capacitance of the Teflon insulation between the cylinders seems to have contributed error, particularly to the dissipation factor results. Consequently a Berberich type, guarded cup cell has been used. In this case the capacitance measurement in the gas cannot be guaranteed because condensation to liquid may have occurred in the cell before it was finally immersed. The values of dissipation factor should, however, be accurate even though the bridge (Wayne-Kerr) sensitivity is not entirely adequate. A new General Radio transformer ratio-arm bridge is expected to be delivered very soon. The sensitivity of this bridge is considerably greater and should permit such measurements with greater precision.

Results are given in Table II.

It is interesting that as expected theoretically, no absorption effects are noticed at least over the limited frequency range used. In view of the recognized inaccuracies, the dielectric constant values check the literature values quite well. Surprisingly no literature reference to dielectric loss has been discovered which makes these measurements particularly important.

PROGRAM FOR OCTOBER AND THE SIXTH QUARTER

In October the work outlined in this report will continue. By the end of the Sixth quarter it is expected that most of the evaluations after aging at 250° C will have been completed. Some of the evaluation of samples aged at 120 in both air and vacuum should be underway.

Some consideration has already been given to methods for determining "crush" resistance of insulated wires at cryogenic temperatures. By the end of the next quarter a method should be finalized.

Consideration of ribbon cable and dielectric tests on cryogenic liquids will continue.

Table II

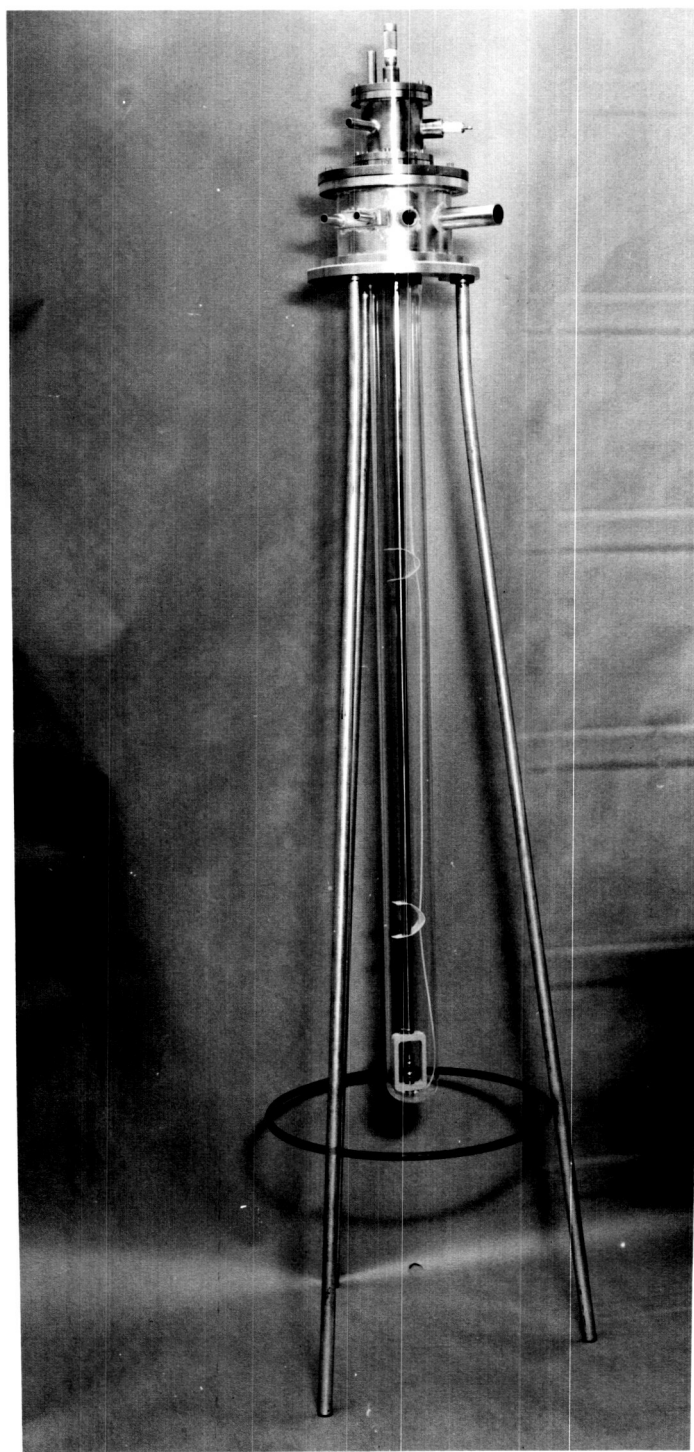
<u>Frequency</u> <u>cps</u>	<u>Dielectric</u> <u>Constant</u>		<u>Dissipation</u> <u>Factor</u>	
	(1)	(2)	(1)	(2)
100	1.040	1.046	.00755	.0053
1000	1.040	1.048	--- *	--- *
10000	1.040	1.046	.00295	.00232
20000	1.040	1.017*	.00373	.00400
Literature Value 1 megacycle ?	1.048		Not given	

* In obvious error

(1) With unguarded cylindrical electrodes

(2) With guarded "Berberich" type cell.

Note: The above values are considered to be representative but undetermined small errors are suspected to exist because of difficulties in measurement of small values at the end of long leads, etc.

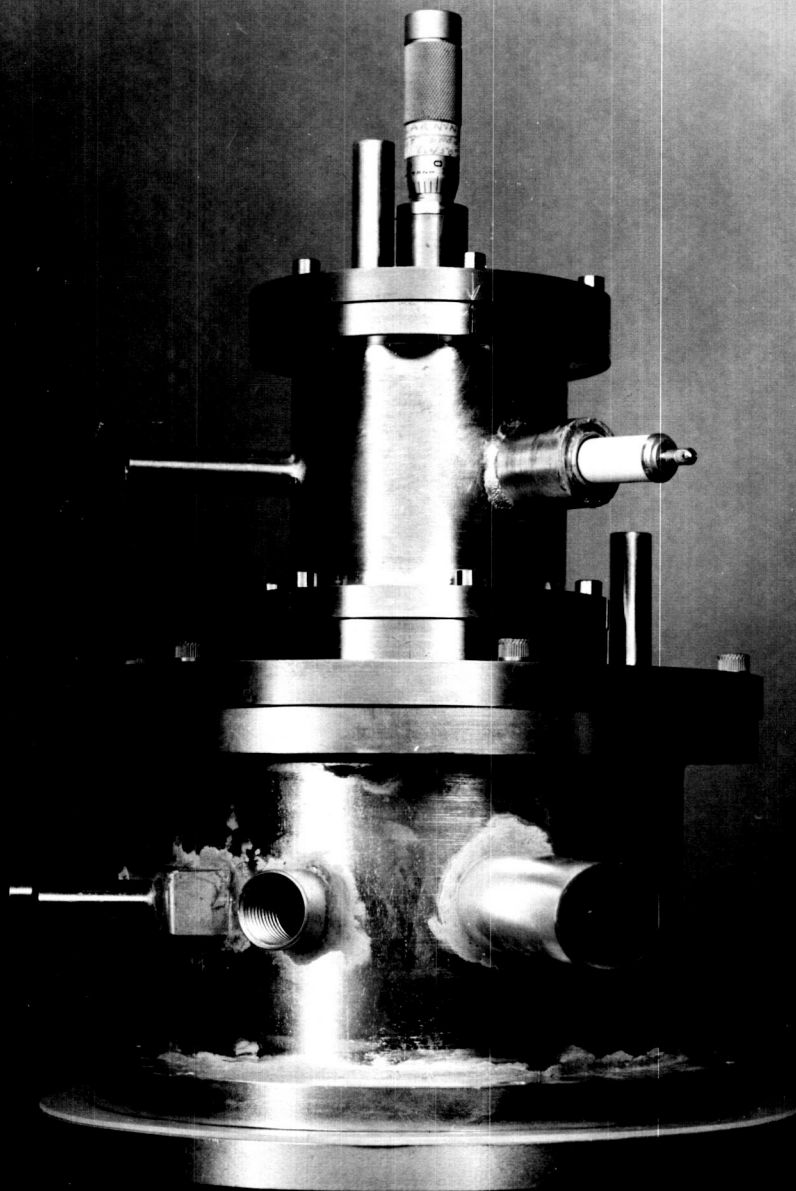


1181 256

ADJUSTABLE SPHERE-GAP BREAKDOWN DEVICE FOR TESTING GASES OR LIQUIFIED GASES.
(PHOTO 2) (CUST. APP. REQ.)

E369.9

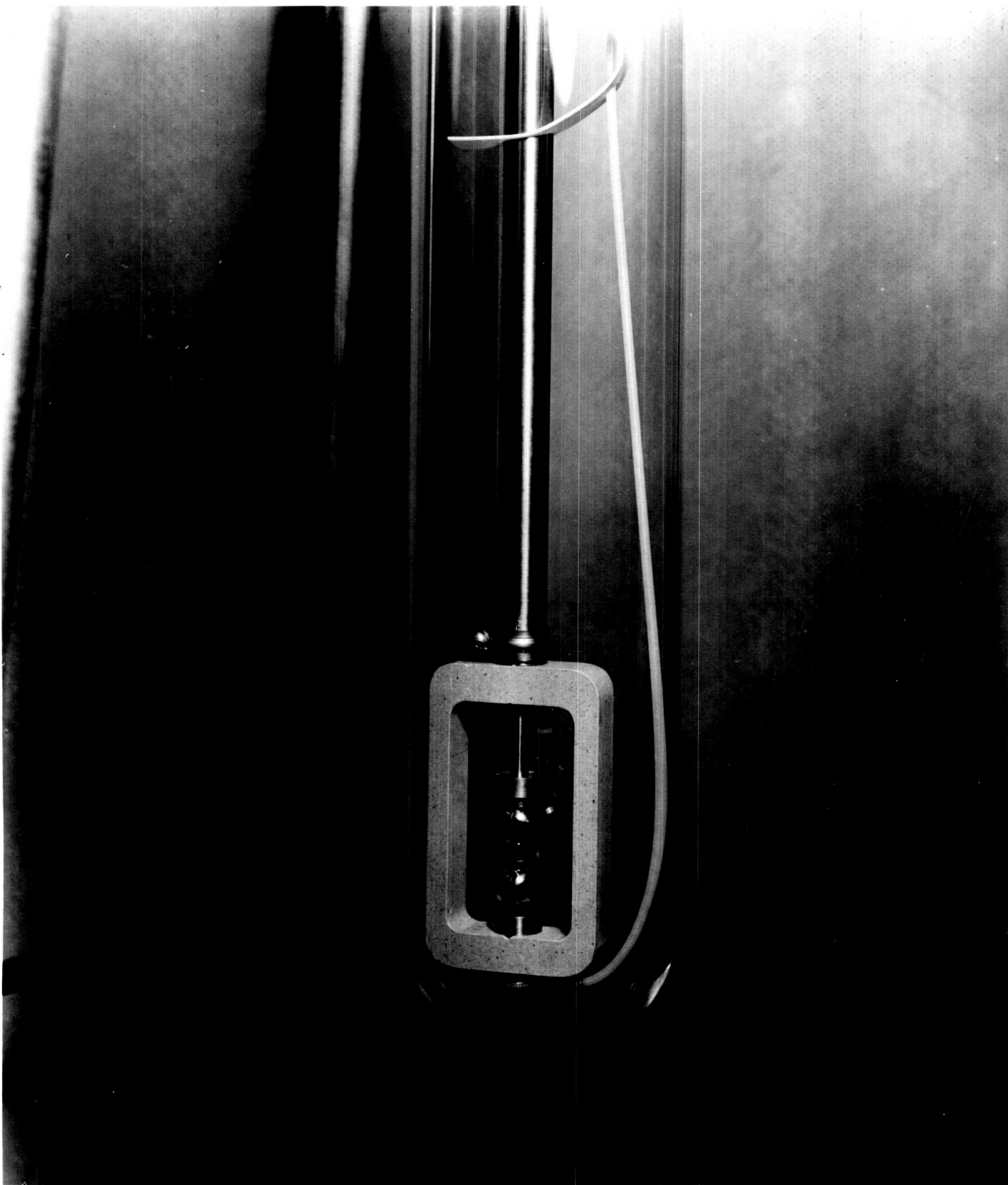
9-25-62



1181 257

HEADER ARRANGEMENT OF SPHERE-GAP BREAKDOWN DEVICE SHOWING MICROMETER ADJUSTMENT AND HERMETICALLY-SEALED HIGH VOLTAGE TERMINAL. (CUST. APP. REQ.)
E369.9

9-25-62



1181 258

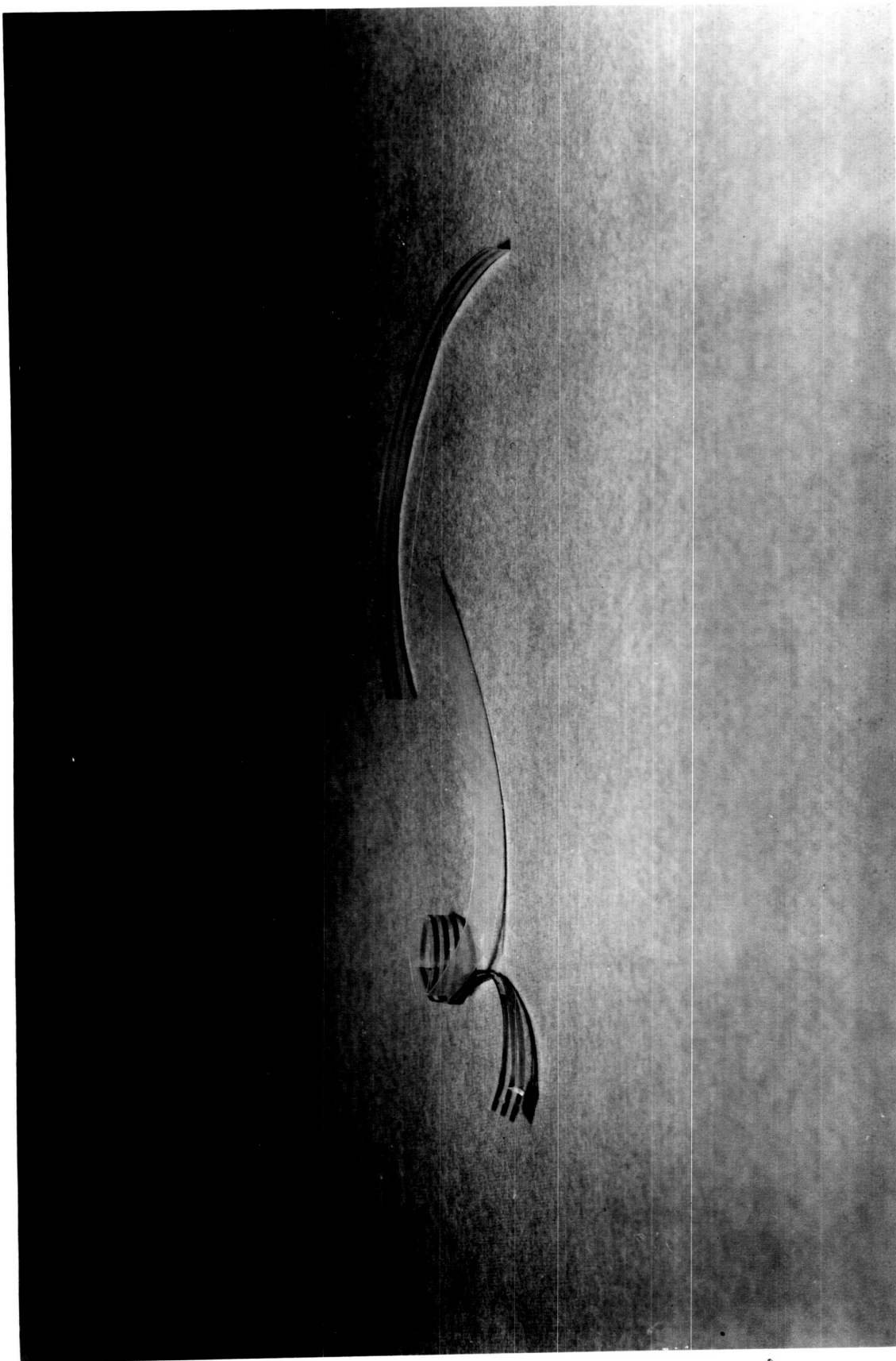
SPHERE-GAP CONSTRUCTION OF BREAKDOWN DEVICE SHOWING MAGNETIC SUPPORTS FOR STEEL
SPHERES IN ONE-PIECE FLUOROSINT HOLDER. (PHOTO 3) (CUST. APP. REQ.)
E369.9

9-25-62



1181 259 INSULATED WIRE DIELECTRIC MEASUREMENTS SPECIMEN. (CUST. APP. REQ.)
E369.4

9-25-62



1181 260

FLAT MULTI-CONDUCTOR RIBBON CABLE AFTER FLEXIBILITY TEST IN LIQUID HELIUM ON
1/4" MANDREL. NOTE THE DELAMINATION OF THE PLASTIC LAYER. UNFLEXED SPECIMEN
IN THE BACKGROUND. (PHOTO 1) (CUST. APP. REQ.) E369.4 9-25-62



1181 359

VACUUM THERMAL AGING EQUIPMENT FOR WIRE TEST. (PHOTO 4) (CUST. APP. REQ.)
E369.4

10-5-62



1181 360

GLASS VACUUM THERMAL AGING TEST CHAMBER SHOWING WIRE SPECIMENS WRAPPED IN
ALUMINUM FOIL. (PHOTO 5) (CUST. APP. REQ.) E369.4 10-5-62